ESRF	**Experiment title:** **Determination of the level of internal stress in samples of TiAl-based alloys after exposure to oxidising atmospheres**	**Experiment number**: MA-679
Beamline: ID31	**Date of experiment**: from: 18/09/08 to: 22/09/08	**Date of report**: 29/06/09
Shifts: 9	**Local contact(s)**: Dr. Alexander EVANS (email: alexander.evans@psi.ch)	*Received at ESRF:*

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Report:

One of the two objectives of the IMPRESS Integrated Project within the EU 6th Framework Program [1] is to develop TiAl materials for use in turbine blade applications. This experimental report forms part of a general work-plan for the IMPRESS Integrated Project as agreed in a Memorandum of Understanding between ESRF, ILL and ESA in January 2008 [2].

TiAl-based alloys have attracted considerable attention as industrial materials for turbine blade applications because of high creep strength, good room-temperature ductility and excellent corrosion resistance. However, these alloys embrittle when exposed to oxidising atmospheres. The consequence of this is that all turbine blades which are to be installed in the near future by both GE and Rolls-Royce will have virtually zero ductility after the engine has been operated for the first time. At present the engines are designed so that the operating stresses are very low so the fact that the blades are embrittled will not detract from the performance of the blades. Nevertheless for the longer term it is important that the origin of the embrittlement is characterised and this is the focus of this experimental report.

Analysis of the surface of samples of Ti48Al2Nb2Cr which have been exposed in air at 700C for 1h has shown that oxygen has diffused in to depths of less than 100nm [3]. Complementary results from Krakow [4] show an oxide layer on Ti46Al8Nb is about 200nm thick after a 2hr exposure at 700ºC and about 1μm thick after a 200hr exposure at 800ºC. The formation of an oxide layer is accompanied by oxygen (nitrogen) penetration of the alloy. It has been assumed that oxygen (nitrogen) diffusion into the surface tries to expand the lattice and the surface is thus put into compression and correspondingly the internal alloy is put under tension. Preliminary measurements using laboratory x-rays have suggested that there is a large residual stress generated in the top 30μm (the approximate penetration depth of the $Cu_{K\alpha}$ used).

During 4 days beamtime on ID31 the $\sin^2\Psi$ method was used in an attempt to characterise the stress on the underlying TiAl-based alloys. Samples were platelets measuring ~1cm^2 and 4mm in thickness. Using a wavelength of λ=0.79999Å the samples were aligned using fluorescent powder. A beamsize of 1.2x150μm was used and therefore the penetration depth is up to 1.2μm depending upon the incident angle of the synchrotron X-rays.

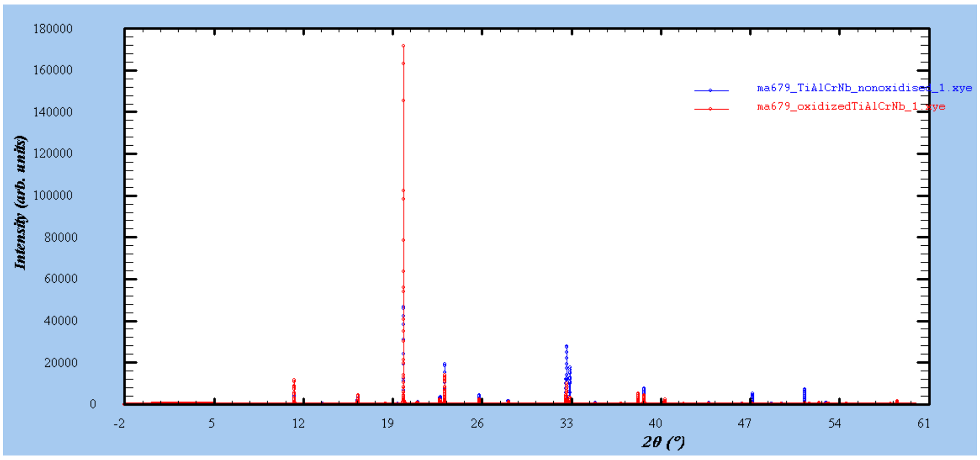

Figure 1. Full diffraction pattern for $Ti_{48}Al_{48}Cr_2Nb_2$ non-oxidised (blue) and oxidised (red).

Large grain sizes within the sample and two main phases with similar lattice parameters generated overlapping bragg peaks, so it was decided to increase the wavelength to λ=1.54Å. At this energy it was possible to see clearer bragg peaks and it was decided to focus on the (222) reflection at 2θ~82°; a prominent peak in the γ-Ti_3Al phase.

In accordance with the $\sin^2\Psi$ method the sample was rotated through the ID31 omega angle of rotation with a range of 40° in 2° steps, whilst the evolution of the bragg peak was obtained for each sample. A list of samples is shown in table 1.

Sample	Coating	Oxidation State
Ti48Al2Cr2Nb	-	Unoxidised
Ti48Al2Cr2Nb	-	Oxidised
Ti46Al8Nb	CrSi	Unoxidised
Ti46Al8Nb	CrSi	Oxidised
Ti46Al8Nb	-	Unoxidised
Ti46Al8Nb	-	Oxidised
Ti46Al8Ta	-	Unoxidised
Ti46Al8Ta	-	Oxidised

Table 1. List of samples measured through 40 degrees of psi at λ=1.54Å.

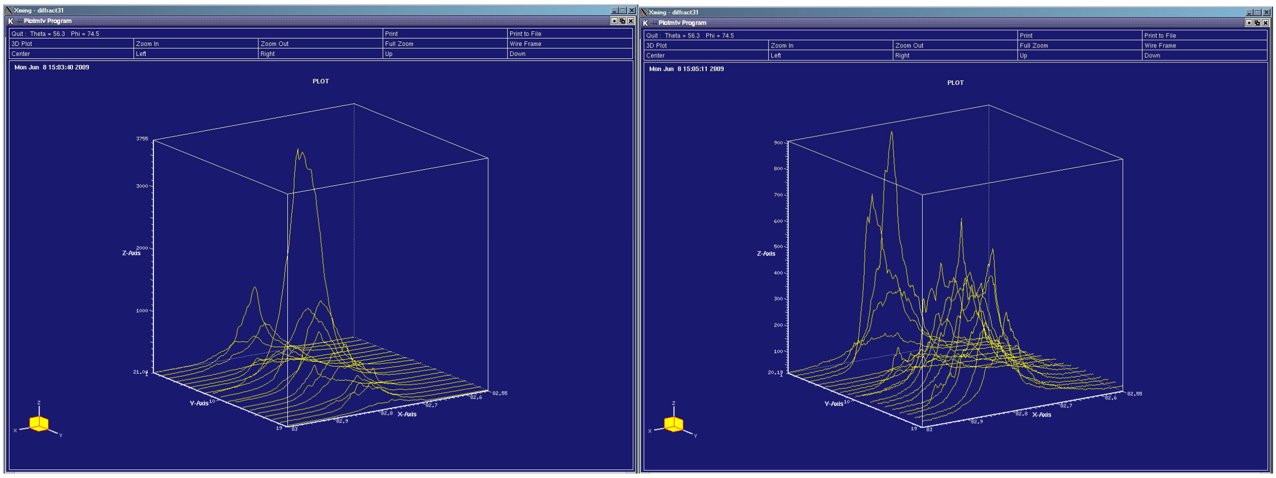

Figure 2. (222) peak through changing Ψ angle for a)$Ti_{46}Al_{46}Nb_8$ and b)oxidised $Ti_{46}Al_{46}Nb_8$.

By obtaining the exact value for 2θ from this data, the associated d-spacing was calculated for each Ψ.

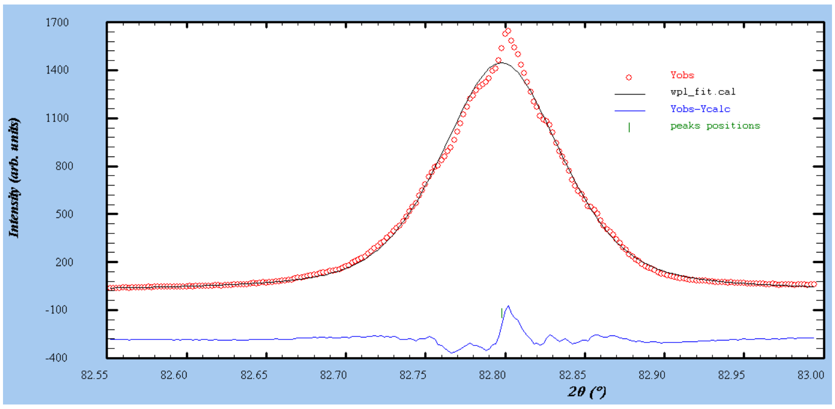

Figure 3. FullProf analytical programme used to fit the (222) peak through changing Ψ angle.

The gradient of the graph of d-spacing against $\sin^2\Psi$ (figure 4) can be put into the equation to calculate stress.

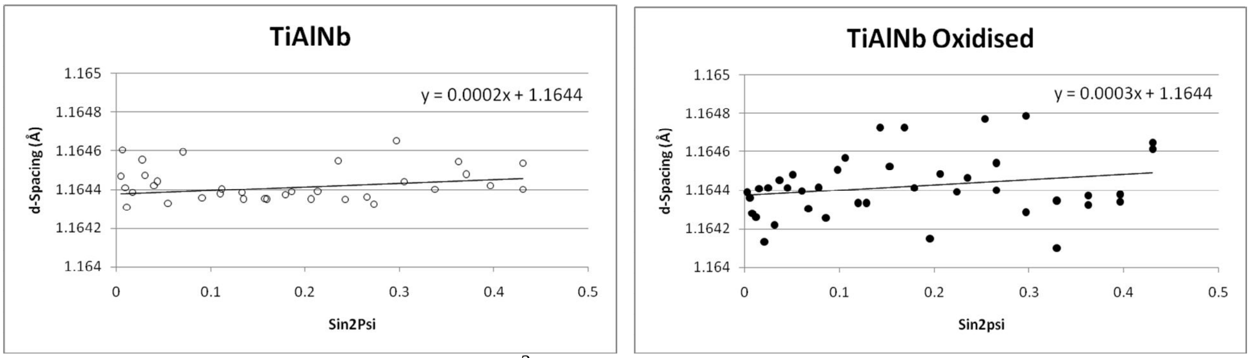

Figure 4. Plots of d-spacing against $\sin^2\Psi$ for the IMPRESS alloy $Ti_{46}Al_{46}Nb_8$.

We suggest that the scatter in parameter measurements is possibly due to segregation leading to different parameters in different regions and that this has masked any stress in the samples. This problem could be overcome by heat-treating the samples for a long time and cooling them very slowly.

References

[1] D. J. Jarvis and D. Voss, Materials Science and Engineering: A, 413-414 (2005) 583
[2] ESA-ESRF-ILL Memorandum of Understanding (2008)
[3] X. Wu, A. Huang, D. Hu, M.H. Loretto, Intermetallics 17 (2009) 540–552
[4] Godlewska, E.; Mitoraj, M.; Morgiel, J., Materials at High Temperatures, Volume 26, Number 1, March 2009 , pp. 99-103(5)